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SEP 76 H P VIND, C W MATHEWS
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FIELD TEST FOR DETECTING LEAD-BASED PAINTS

By

H. P. Vind and C. W. Mathews

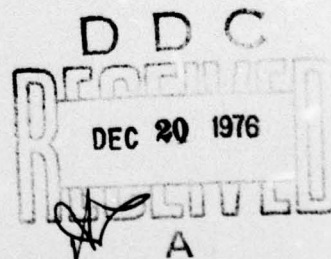
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thickening agent to simplify its application and improve its ability to adhere to walls and ceilings. In CEL tests with the modified sodium sulfide reagent, all the lead compounds tested gave positive results. Conveniently, the minimum concentration of lead that could be detected was approximately equal to the maximum legal limit of 0.5%. Nearly all of the other inorganic compounds commonly employed in the paint formulations reacted negatively. Exceptions were a few biocides and driers usually employed in paint in concentrations too low to interfere with the test for lead.



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H. P. Vind and C. W. Mathews
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A field method for determining whether the lead content of a paint film exceeds the 0.5% limit imposed by federal law and by NAVFAC Instructions appears to be partially fulfilled by a simple spot test introduced at the University of Rochester, New York. In the test, a drop of sodium sulfide solution is placed on a chip of paint and layers of paint that contain lead turn black or gray. The Civil Engineering Laboratory (CEL) modified the lead-indicating reagent by adding solvents to improve its ability to penetrate paint and a thickening agent to simplify its application and improve its ability to adhere to walls and ceilings. In CEL tests with the modified sodium sulfide reagent, all the lead compounds tested gave positive results. Conveniently, the minimum concentration of lead that could be detected was approximately equal to the maximum legal limit of 0.5%. Nearly all of the other inorganic compounds commonly employed in the paint formulations reacted negatively. Exceptions were a few biocides and driers usually employed in paint in concentrations too low to interfere with the test for lead.

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INTRODUCTION

In Naval Facilities Engineering Command (NAVFAC) Instruction 10365.2A, Navy activities are directed to insure that the lead content of paint applied to barracks and dependents' quarters does not exceed 0.5% of the weight of the dried film. The activities are also directed to remove or cover previously applied paint if it constitutes a serious lead-poisoning hazard. The instruction is in compliance with Public Laws 91-695 and 93-151. A field method for determining whether the lead content of a paint film exceeds 0.5% is needed to implement the instruction. This technical note deals only with an investigation of a chemical spot test. A report on investigations of other methods will be issued later.

REAGENT FOR DETECTING LEAD IN PAINT

An inexpensive and easily used reagent for detecting lead in paint was introduced by Professors J.W. Sayre and D.W. Wilson [1,2,3] at the University of Rochester, New York. They placed a drop of an aqueous sodium sulfide solution (5 to 8%) on a chip of paint. Layers of paint that contained lead turned black or gray: the greater the concentration of lead, the darker the gray. The Navy has employed this reagent in a kit for identifying samples of weathered paint in the field [4].

The sodium sulfide reagent was recently modified at the Civil Engineering Laboratory (CEL) by thickening it with methyl cellulose (approximately 3%). Thickening made the reagent easier to apply and improved its ability to adhere to walls and ceilings. Also, thickened reagent can be applied directly to a paint chip, thereby eliminating the need for a spot plate. (When a chip of paint is to be examined and when a spot plate, hand lens, and tweezers are available, the unthickened reagent as originally specified by Sayre and Wilson is perhaps superior to the thickened reagent, because it more readily wets the paint chips.)

The reagent was further modified at CEL by addition of paint-removing solvents. One or two ml each of methyl ethyl ketone, 1,1,1-trichloroethane and 2-methyl-1-pyrrolidinone were added per 100 ml of reagent. The solvents were added to help the reagent penetrate into a paint film.

Both the thickened and unthickened reagents remain sensitive to lead for many months in well-stoppered containers. If the containers are not covered, the reagents rapidly lose hydrogen sulfide and become insensitive to lead. Sensitivity of the reagents can be tested by

applying them to strips of lead acetate paper or to chips of paint known to contain lead. The reagents keep longer if they are refrigerated when not in use.

The small quantities of sodium sulfide reagent contained in dropping bottles, the customary containers, are no more dangerous to handle than equivalent amounts of diluted lye water. Nevertheless, the reagents are strongly alkaline and should be kept out of the eyes and mouth and out of the reach of children. Skin irritation by the reagents is minimal, but the reagents will cause chapped hands if not promptly washed off with water.

LEAD-IN-PAINT DETECTOR KITS

Tiny "vest pocket" lead-in-paint detector kits (Figure 1) were assembled. Each kit contains a 15-ml dropping bottle filled with the thickened sodium sulfide reagent, a sharpened nail, and a few cotton-tipped applicator sticks. The reagent can be applied to a painted surface or to a paint chip either directly from the dropping bottles, or it can be applied with clean cotton-tipped applicator sticks. Even though the reagent contains paint remover solvents, it will not penetrate underlying layers of paint and primer coats. Therefore, a pair of 1/2-inch-long crossed scratches should be scribed by the sharpened nail completely through the paint to the underlying substrate. The reagent can be applied at the point of intersection of the scratches. The effect of the reagent on all layers of paint can then be observed.

The effect of the reagent on the paint can best be observed with the aid of a hand lens, such as that supplied with the kit shown in Figure 2. This kit is more complete than the first. Besides the illuminated hand lens, it contains a spot plate, knife, tweezers, wash bottle, sharpened nail, and bottles of thickened and unthickened sodium sulfide reagents. It also contains reference samples of paint known to contain lead; and it contains envelopes for inserting samples of paint scrapings.

SENSITIVITY OF THE SODIUM SULFIDE TEST

Tests were made of the sensitivity of the sodium sulfide reagents to various lead compounds. All of those lead compounds that were not already black turned black or very dark when they were wetted with either of the sodium sulfide reagents. The compounds tested included the acetate, carbonate, chromate, resinate, stannate, and sulfate of lead and both red and yellow lead oxides. Lead dioxide, lead sulfide, and lead phthalocyanine are already black or very dark and remained so when wetted with the sulfide reagents.



Figure 1. 'Vest pocket' lead-in-paint detector kit.

Conveniently, approximately 0.5% was the minimum concentration of lead that could be detected in light-colored paints by the sodium sulfide reagent (Figure 3). The minimum concentration that could be detected in dark-colored paints would no doubt be greater than 0.5%; and it would obviously be impossible to detect lead in black paints by means of the sodium sulfide reagent. Fortunately, black paints are usually pigmented with carbon black rather than with lead compounds. Black paints usually contain only those lead compounds employed as driers or as agents to prevent corrosion.

SPECIFICITY OF THE SODIUM SULFIDE TEST

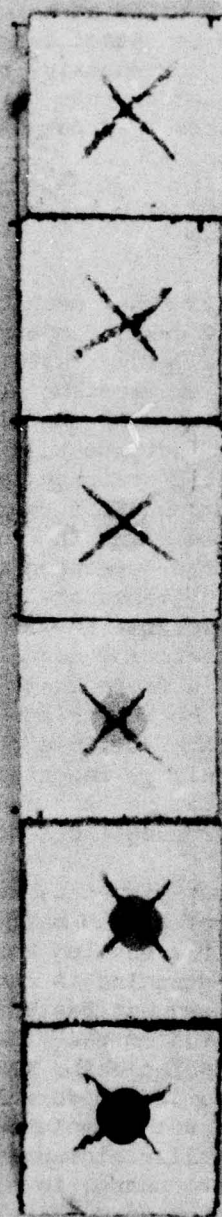
In addition to lead, there are 13 other metallic elements (Table 1) whose sulfides might be black or very dark. Compounds of all but two of them have been employed in paint as pigments, driers, biocides, or corrosion inhibitors. Thus, it might at first be concluded that the sodium sulfide test is too nonspecific to be useful.

However, further considerations indicate that the sodium sulfide test may be fairly specific for paint containing lead. Except for pigments containing lead or iron, compounds of the elements listed in Table 1 are not frequently employed in paint in concentrations sufficiently high to interfere with the detection of lead at concentrations of 0.5% and greater. The iron pigments are not likely to interfere with the test because they are so insoluble in water they are unlikely to react with aqueous reagents. Furthermore, not all of the sulfides of all of the metallic elements listed in Table 1 are black. There is only one sulfide of lead and it is black, but there are as many as five sulfides of some of the elements. Black sulfides of the other elements of Table 1 are likely to be formed only by reaction of sodium sulfide with those compounds in which the metal is oxidized to a specific valence state. Sodium sulfide will reduce the ions of some metals to the lowest valence state.

In tests at CEL the popular paint pigments, red and yellow iron oxides, were wetted with a solution of sodium sulfide. Neither of the iron compounds changed color during the ensuing hour. However, the yellow iron oxide did turn black on standing in contact with the reagent over the weekend. In all subsequent tests at CEL, observations of color were made within a few minutes of the time that the reagent was applied to the test compound or paint chip. Ferric (iron) chloride, a water-soluble compound not employed in the formulation of paints, turned black immediately when wetted with a solution of sodium sulfide.

Numerous other compounds of metallic elements whose sulfides may be black, or very dark, also failed to change to a darker color when they were wetted with a solution of sodium sulfide. Compounds exhibiting such behavior included antimony oxide, antimony trisulfide, bismuth trioxide, bismuth subcarbonate, calcium chromate, chromic oxide, chromic hydroxide, cuprous oxide, manganous sulfate, red mercuric oxide, molybdic acid, and nickel stannate.

SODIUM SULFIDE TEST FOR LEAD IN PAINT*



Lead Content of Dried Paint Film	
50.4%	12.5%
	2.5%
	0.5%
	0.1%
	0.0%

*Aqueous mixture of 3% sodium sulfide and 3% methyl cellulose
with paint-remover solvents added

Figure 3. Spots developed by action of sodium sulfide reagent in paints containing various concentrations of lead.

Table 1. Metallic Elements Having at Least One Black Sulfide

Element	Colors of Sulfides	Some Uses of Compounds in Paints
Actinium	black	None
Antimony	black, red	Pigment
Bismuth	black, brown, gray	Pigment
Chromium	black, brown	Pigment, corrosion inhibitor
Cobalt	black, gray, red	Pigment, drier
Copper	black	Biocidal pigment
Iron	black, green, yellow	Pigment
Lead	black	Pigment, drier, corrosion inhibitor
Manganese	black, green, pink	Pigment, drier
Mercury	black, red	Pigment, biocide
Molybdenum	black, brown, gray	Pigment, corrosion inhibitor
Nickel	black, gray, yellow	Pigment
Silver	black, gray	None
Tin	black, gold, gray	Gilding agent

Inorganic compounds containing metallic elements whose sulfides are not black would not be expected to turn black on contact with a sodium sulfide reagent. Nevertheless, several such compounds that might be employed in the formulation of paints were tested. The compounds or materials included aluminum oxide, arsenious oxide, barium monoxide, barium dioxide, barium sulfate, beryllium oxide, cadmium chromate, calcium carbonate, magnesium sulfate, silicic acid, titanium dioxide, zinc oxide, talc, and infusorial earth. As would be expected, none of them turned dark in contact with the sodium sulfide reagent.

Numerous compounds turn black in the presence of sodium sulfide even though they contain no lead - such as ferric chloride, which is not employed in the formulation of paints. In the tests at CEL, mercuric oxide, mercuric iodide, and phenylmercuric oleate also turned black; they are frequently employed in paints as biocides. (As noted previously, the pigment, red mercuric sulfide, is not converted to a black sulfide by the sodium sulfide reagent.) Cobalt naphthenate and manganese naphthenate, two compounds frequently employed as drying or curing agents in paints, both turned black when contacted by the sulfide reagent. Biocides and drying agents are employed very widely in the formulation of paints, but are not normally employed in high enough concentrations to interfere with the test for lead. Bismuth trioxide might cause some trouble because it changed from a greenish white to a light brown in the presence of the sulfide reagent.

CONCLUSIONS

The sodium sulfide test appears to be reasonably specific for lead when the testing is restricted to paints, especially interior and exterior house paints. All light-colored paints containing approximately 0.5% or more lead will be detected.

RECOMMENDATIONS

On the basis of these investigations, the sodium sulfide test is recommended for use to detect lead-containing paints. The test is sensitive, reasonably specific, very inexpensive, and easy to perform.

It is especially recommended that the sodium sulfide test be employed whenever new paint has been applied to residential structures. It is the only test that can be used for this purpose because X-ray instruments respond to lead in underlying paint as well as to the surface layer. Furthermore, instruments are not sufficiently sensitive to detect lead in a single layer of paint unless the paint has a lead content of at least about 10 times the legal limit. If the test is

performed on a test strip before the painting begins, it will be possible to ascertain if the paint is acceptable in time to prevent the need for costly corrective measures. Strict enforcement of the specifications regarding the lead content of new paint will eventually eliminate the lead paint hazard in Navy housing.

It is also recommended that the sodium sulfide test be employed to determine whether underlying layers of older paint constitute a lead paint hazard. X-ray instruments are a useful supplement for this. Rather than indicating the concentrations of lead in paint films, the instruments indicate the total quantities of lead per unit area in all layers of paint. The instruments are therefore useful for locating hazardous accumulations of lead in paint, but the sodium sulfide test is needed to determine whether the concentration of lead in any of the layers of paint exceeds the legal limit.

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